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N-(3-Chlorophenyl)-2-nitrobenzenesulfonamide

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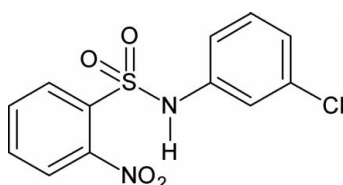
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.039; wR factor = 0.100; data-to-parameter ratio = 14.7.

In the title compound, $\text{C}_{12}\text{H}_9\text{ClN}_2\text{O}_4\text{S}$, the dihedral angle between the aromatic rings is $73.65(7)^\circ$. The amide H atom shows bifurcated hydrogen bonding, generating an intramolecular $S(7)$ and an intermolecular $C(4)$ motif.

Related literature

For studies on the effects of substituents on the structures and other aspects of N -(aryl)-amides, see: Alkan *et al.* (2011); Bowes *et al.* (2003); Gowda *et al.* (2000); Saeed *et al.* (2010); Shahwar *et al.* (2012), of N -aroylsulfonamides, see: Suchetan *et al.* (2012), of N -arylsulfonamides, see: Gowda *et al.* (2002) and of N -chloroarylsulfonamides, see: Gowda & Shetty (2004); Shetty & Gowda (2004). For hydrogen-bonding patterns and motifs, see: Adsmund *et al.* (2001); Allen *et al.* (1998); Bernstein *et al.* (1995); Etter (1990).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_9\text{ClN}_2\text{O}_4\text{S}$ $M_r = 312.72$ Monoclinic, $P2_1/c$ $a = 8.9856(7)$ Å $b = 9.5794(8)$ Å $c = 15.796(1)$ Å $\beta = 103.120(8)^\circ$ $V = 1324.17(17)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.46$ mm⁻¹ $T = 293$ K $0.48 \times 0.48 \times 0.24$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector

Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009)

 $T_{\min} = 0.809$, $T_{\max} = 0.898$

5230 measured reflections

2702 independent reflections

2201 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.013$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.039$ $wR(F^2) = 0.100$ $S = 1.03$

2702 reflections

184 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 0.39$ e Å⁻³ $\Delta\rho_{\min} = -0.51$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O3$	0.82 (2)	2.33 (2)	2.864 (2)	123 (2)
$N1-H1N\cdots O2^i$	0.82 (2)	2.33 (2)	3.061 (2)	148 (2)

Symmetry code: (i) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5982).

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supplementary materials

Acta Cryst. (2012). E68, o2576 [doi:10.1107/S1600536812033272]

***N*-(3-Chlorophenyl)-2-nitrobenzenesulfonamide**

U. Chaithanya, Sabine Foro and B. Thimme Gowda

Comment

As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Alkan *et al.*, 2011; Bowes *et al.*, 2003; Gowda *et al.*, 2000; Saeed *et al.*, 2010; Shahwar *et al.*, 2012); *N*-arylsulfonamides (Suchetan *et al.*, 2012); *N*-arylsulfonamides (Gowda *et al.*, 2002) and *N*-chloroarylsulfonamides (Gowda & Shetty, 2004; Shetty & Gowda, 2004), in the present work, the crystal structure of *N*-(3-Chlorophenyl)-2-nitrobenzenesulfonamide has been determined (Fig. 1).

The conformation of the N—C bond in the —SO₂—NH—C segment has *gauche* torsion with respect to the S=O bonds (Fig.1), similar to that observed in *N*-(3-chlorobenzoyl)-2-nitrobenzenesulfonamide (I) (Suchetan *et al.*, 2012). Further, the conformation of the N—H bond in the —SO₂—NH— segment is *syn* to both the *ortho*-nitro group in the sulfonyl benzene ring and *meta*-Cl atom in the anilino ring. The molecule is twisted at the S—N bond with the torsional angle of 48.46 (18)°, compared to the value of 65.41 (38)° in (I).

The dihedral angle between the sulfonyl and the anilino rings is 73.65 (7)°, compared to the value of 89.1 (1)° in (I).

The amide H-atom showed bifurcated intramolecular H-bonding with the O-atom of the *ortho*-nitro group in the sulfonyl benzene ring and the intermolecular H-bonding with the sulfonyl oxygen atom of the other molecule, generating S(7) and C(4) motifs (Adsmund *et al.*, 2001; Allen *et al.*, 1998; Bernstein *et al.*, 1995; Etter, 1990).

In the crystal, the intermolecular N—H···O (S) hydrogen bonds (Table 1) link the molecules into chains. Part of the crystal structure is shown in Fig. 2.

Experimental

The title compound was prepared by treating 2-nitrobenzenesulfonylchloride with 3-chloroaniline in the stoichiometric ratio and boiling the reaction mixture for 15 minutes. The reaction mixture was then cooled to room temperature and added to ice cold water (100 ml). The resultant solid *N*-(3-chlorophenyl)-2-nitrobenzenesulfonamide was filtered under suction and washed thoroughly with cold water and dilute HCl to remove the excess sulfonylchloride and aniline, respectively. It was then recrystallized to constant melting point from dilute ethanol. The purity of the compound was checked and characterized by its infrared spectra.

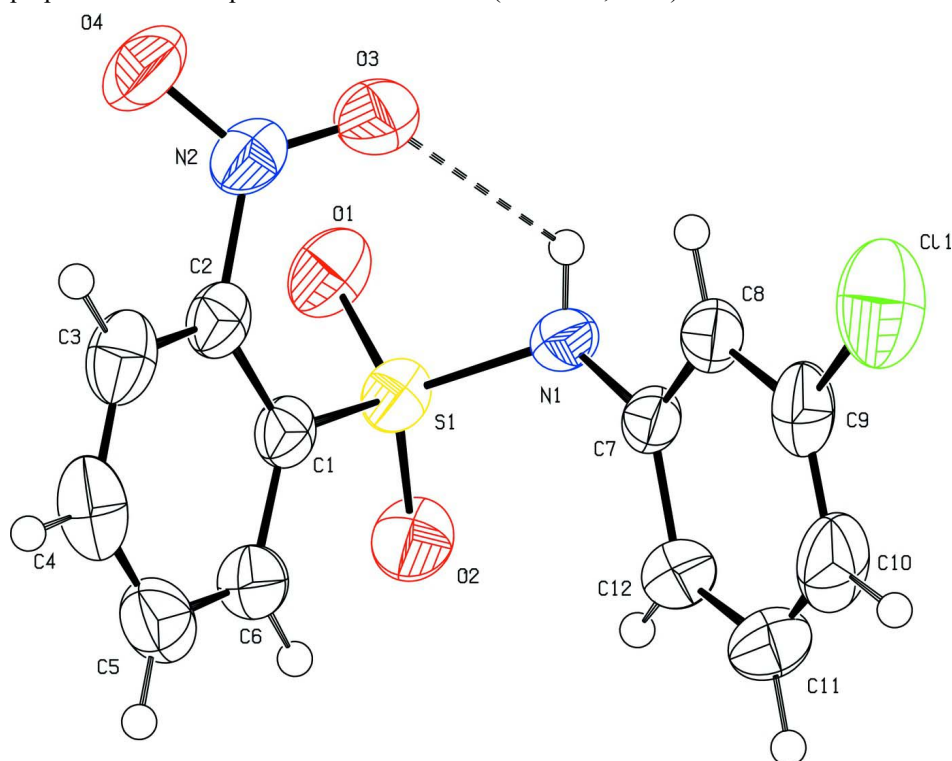
Rod like colourless single crystals of the title compound used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation of the solvent at room temperature.

Refinement

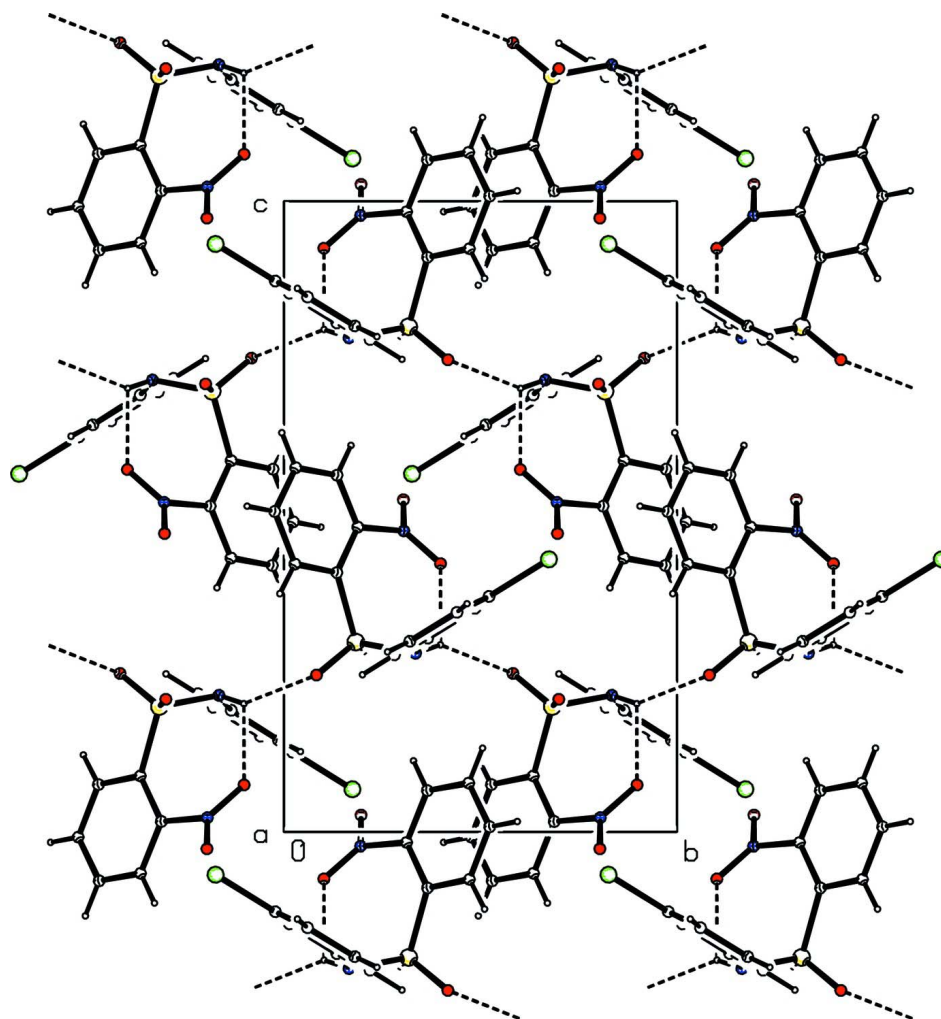
H atoms bonded to C were positioned with idealized geometry using a riding model with C—H = 0.93 Å. The amino H atom was freely refined with the N—H distance restrained to 0.86 (2) Å. All H atoms were refined with isotropic displacement parameters set to 1.2 *U*_{eq} of the parent atom.

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

**Figure 1**

Molecular structure of the title compound, showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level.

**Figure 2**

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

***N*-(3-Chlorophenyl)-2-nitrobenzenesulfonamide**

Crystal data

$C_{12}H_9ClN_2O_4S$

$M_r = 312.72$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 8.9856\ (7)\ \text{\AA}$

$b = 9.5794\ (8)\ \text{\AA}$

$c = 15.796\ (1)\ \text{\AA}$

$\beta = 103.120\ (8)^\circ$

$V = 1324.17\ (17)\ \text{\AA}^3$

$Z = 4$

$F(000) = 640$

$D_x = 1.569\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2323 reflections

$\theta = 2.6\text{--}27.8^\circ$

$\mu = 0.46\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Rod, colourless

$0.48 \times 0.48 \times 0.24\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur

diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube

Graphite monochromator

Rotation method data acquisition using ω scans

Absorption correction: multi-scan
(*CrysAlis RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.809$, $T_{\max} = 0.898$
5230 measured reflections
2702 independent reflections
2201 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.013$
 $\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.7^\circ$
 $h = -11 \rightarrow 11$
 $k = -11 \rightarrow 10$
 $l = -19 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.100$
 $S = 1.03$
2702 reflections
184 parameters
1 restraint
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.7223P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.51 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. Absorption correction: *CrysAlis RED* (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in *SCALE3 ABSPACK* scaling algorithm.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1289 (2)	0.1345 (2)	0.41117 (13)	0.0364 (4)
C2	0.0829 (2)	0.1878 (2)	0.48339 (13)	0.0395 (5)
C3	0.1398 (3)	0.1350 (3)	0.56563 (14)	0.0523 (6)
H3	0.1062	0.1705	0.6128	0.063*
C4	0.2467 (3)	0.0298 (3)	0.57749 (17)	0.0606 (7)
H4	0.2859	−0.0054	0.6329	0.073*
C5	0.2954 (3)	−0.0235 (3)	0.50813 (18)	0.0578 (6)
H5	0.3687	−0.0938	0.5165	0.069*
C6	0.2350 (3)	0.0278 (2)	0.42487 (16)	0.0487 (5)
H6	0.2667	−0.0105	0.3778	0.058*
C7	0.2763 (2)	0.3656 (2)	0.30709 (12)	0.0350 (4)
C8	0.3206 (2)	0.4862 (2)	0.35429 (13)	0.0403 (5)
H8	0.2492	0.5418	0.3726	0.048*
C9	0.4733 (3)	0.5219 (3)	0.37354 (15)	0.0518 (6)
C10	0.5814 (3)	0.4401 (3)	0.34821 (19)	0.0673 (8)
H10	0.6839	0.4657	0.3619	0.081*
C11	0.5348 (3)	0.3200 (3)	0.3024 (2)	0.0681 (8)

H11	0.6072	0.2635	0.2855	0.082*
C12	0.3822 (3)	0.2810 (3)	0.28080 (16)	0.0518 (6)
H12	0.3518	0.1998	0.2493	0.062*
N1	0.11632 (18)	0.33458 (18)	0.28331 (11)	0.0370 (4)
H1N	0.064 (2)	0.398 (2)	0.2966 (14)	0.044*
N2	−0.0256 (2)	0.3049 (2)	0.47737 (12)	0.0466 (5)
O1	−0.11286 (16)	0.19931 (18)	0.28970 (10)	0.0530 (4)
O2	0.10397 (19)	0.08275 (17)	0.24876 (10)	0.0543 (4)
O3	−0.02102 (19)	0.39843 (19)	0.42550 (11)	0.0568 (4)
O4	−0.1124 (2)	0.3039 (2)	0.52670 (14)	0.0756 (6)
Cl1	0.52921 (10)	0.67414 (9)	0.43224 (5)	0.0871 (3)
S1	0.04801 (6)	0.18286 (6)	0.30104 (3)	0.03894 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0351 (10)	0.0342 (10)	0.0412 (10)	−0.0101 (8)	0.0112 (8)	−0.0039 (8)
C2	0.0373 (10)	0.0400 (12)	0.0428 (11)	−0.0128 (9)	0.0122 (8)	−0.0069 (9)
C3	0.0558 (14)	0.0600 (15)	0.0418 (12)	−0.0198 (12)	0.0128 (10)	−0.0053 (11)
C4	0.0624 (16)	0.0595 (16)	0.0550 (14)	−0.0185 (13)	0.0031 (12)	0.0140 (12)
C5	0.0468 (13)	0.0469 (14)	0.0770 (18)	−0.0033 (11)	0.0087 (12)	0.0124 (13)
C6	0.0481 (12)	0.0412 (12)	0.0606 (14)	−0.0033 (10)	0.0200 (10)	−0.0003 (11)
C7	0.0323 (10)	0.0396 (11)	0.0348 (10)	0.0003 (8)	0.0110 (8)	0.0069 (8)
C8	0.0388 (11)	0.0426 (12)	0.0409 (11)	−0.0022 (9)	0.0118 (8)	0.0069 (9)
C9	0.0441 (12)	0.0574 (15)	0.0518 (13)	−0.0154 (11)	0.0068 (10)	0.0139 (11)
C10	0.0340 (12)	0.080 (2)	0.0860 (19)	−0.0089 (13)	0.0097 (12)	0.0291 (16)
C11	0.0415 (13)	0.0696 (19)	0.100 (2)	0.0161 (13)	0.0311 (14)	0.0207 (16)
C12	0.0445 (12)	0.0476 (14)	0.0681 (15)	0.0053 (10)	0.0232 (11)	0.0053 (11)
N1	0.0313 (9)	0.0385 (10)	0.0425 (9)	0.0017 (7)	0.0111 (7)	−0.0010 (7)
N2	0.0455 (10)	0.0482 (11)	0.0501 (11)	−0.0116 (9)	0.0193 (8)	−0.0181 (9)
O1	0.0340 (8)	0.0703 (11)	0.0527 (9)	−0.0150 (7)	0.0058 (7)	−0.0102 (8)
O2	0.0677 (10)	0.0496 (10)	0.0501 (9)	−0.0113 (8)	0.0227 (8)	−0.0193 (7)
O3	0.0660 (11)	0.0543 (10)	0.0548 (10)	0.0076 (8)	0.0234 (8)	−0.0031 (8)
O4	0.0798 (13)	0.0675 (12)	0.1002 (15)	−0.0106 (10)	0.0640 (12)	−0.0179 (11)
Cl1	0.0842 (5)	0.0888 (6)	0.0840 (5)	−0.0501 (4)	0.0103 (4)	−0.0107 (4)
S1	0.0383 (3)	0.0421 (3)	0.0372 (3)	−0.0093 (2)	0.0102 (2)	−0.0102 (2)

Geometric parameters (Å, °)

C1—C6	1.381 (3)	C8—C9	1.379 (3)
C1—C2	1.395 (3)	C8—H8	0.9300
C1—S1	1.788 (2)	C9—C10	1.376 (4)
C2—C3	1.380 (3)	C9—Cl1	1.740 (3)
C2—N2	1.475 (3)	C10—C11	1.374 (4)
C3—C4	1.376 (4)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.387 (3)
C4—C5	1.368 (4)	C11—H11	0.9300
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.393 (3)	N1—S1	1.6265 (18)
C5—H5	0.9300	N1—H1N	0.823 (16)

C6—H6	0.9300	N2—O3	1.221 (2)
C7—C8	1.383 (3)	N2—O4	1.222 (2)
C7—C12	1.384 (3)	O1—S1	1.4243 (15)
C7—N1	1.432 (2)	O2—S1	1.4294 (16)
C6—C1—C2	117.7 (2)	C10—C9—C8	121.8 (2)
C6—C1—S1	117.27 (16)	C10—C9—C11	119.61 (19)
C2—C1—S1	124.76 (16)	C8—C9—C11	118.6 (2)
C3—C2—C1	121.3 (2)	C11—C10—C9	118.6 (2)
C3—C2—N2	116.0 (2)	C11—C10—H10	120.7
C1—C2—N2	122.67 (19)	C9—C10—H10	120.7
C4—C3—C2	119.7 (2)	C10—C11—C12	121.4 (2)
C4—C3—H3	120.2	C10—C11—H11	119.3
C2—C3—H3	120.2	C12—C11—H11	119.3
C5—C4—C3	120.3 (2)	C7—C12—C11	118.5 (2)
C5—C4—H4	119.8	C7—C12—H12	120.7
C3—C4—H4	119.8	C11—C12—H12	120.7
C4—C5—C6	119.8 (2)	C7—N1—S1	122.41 (14)
C4—C5—H5	120.1	C7—N1—H1N	112.1 (16)
C6—C5—H5	120.1	S1—N1—H1N	110.8 (16)
C1—C6—C5	121.1 (2)	O3—N2—O4	123.9 (2)
C1—C6—H6	119.5	O3—N2—C2	118.63 (17)
C5—C6—H6	119.5	O4—N2—C2	117.5 (2)
C8—C7—C12	121.12 (19)	O1—S1—O2	118.79 (10)
C8—C7—N1	117.65 (18)	O1—S1—N1	106.94 (10)
C12—C7—N1	121.2 (2)	O2—S1—N1	107.70 (9)
C9—C8—C7	118.5 (2)	O1—S1—C1	109.15 (9)
C9—C8—H8	120.8	O2—S1—C1	105.64 (10)
C7—C8—H8	120.8	N1—S1—C1	108.25 (9)
C6—C1—C2—C3	−0.7 (3)	C8—C7—C12—C11	−0.3 (3)
S1—C1—C2—C3	173.26 (16)	N1—C7—C12—C11	177.0 (2)
C6—C1—C2—N2	177.83 (18)	C10—C11—C12—C7	−0.5 (4)
S1—C1—C2—N2	−8.2 (3)	C8—C7—N1—S1	−129.76 (17)
C1—C2—C3—C4	1.4 (3)	C12—C7—N1—S1	52.8 (2)
N2—C2—C3—C4	−177.20 (19)	C3—C2—N2—O3	142.9 (2)
C2—C3—C4—C5	−0.6 (3)	C1—C2—N2—O3	−35.7 (3)
C3—C4—C5—C6	−0.9 (4)	C3—C2—N2—O4	−35.2 (3)
C2—C1—C6—C5	−0.8 (3)	C1—C2—N2—O4	146.2 (2)
S1—C1—C6—C5	−175.25 (17)	C7—N1—S1—O1	165.95 (15)
C4—C5—C6—C1	1.6 (3)	C7—N1—S1—O2	−65.32 (17)
C12—C7—C8—C9	1.1 (3)	C7—N1—S1—C1	48.46 (18)
N1—C7—C8—C9	−176.38 (18)	C6—C1—S1—O1	135.23 (16)
C7—C8—C9—C10	−1.0 (3)	C2—C1—S1—O1	−38.75 (19)
C7—C8—C9—C11	179.54 (15)	C6—C1—S1—O2	6.42 (18)
C8—C9—C10—C11	0.1 (4)	C2—C1—S1—O2	−167.56 (16)
C11—C9—C10—C11	179.6 (2)	C6—C1—S1—N1	−108.71 (17)
C9—C10—C11—C12	0.6 (4)	C2—C1—S1—N1	77.30 (18)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots O3	0.82 (2)	2.33 (2)	2.864 (2)	123 (2)
N1—H1N \cdots O2 ⁱ	0.82 (2)	2.33 (2)	3.061 (2)	148 (2)

Symmetry code: (i) $-x, y+1/2, -z+1/2$.